APPENDIX I

SAMPLING PROTOCOL

Cal/EPA
California
Environmental
Protection
Agency

Air Resources Board
P.O. Box 2815
2020 L Street
Sacramento, CA
95812-2815

MEMORANDUM

Pete Wilson
Governor

James M. Strock
Secretary for
Environmental
Protection

TO: John S. Sanders, Ph.D., Chief
Environmental Monitoring and Pest
Management Branch
Department of Pesticide Regulation

FROM: George Lew, Chief
Engineering and Laboratory Branch

DATE: January 10, 1997

SUBJECT: FINAL PROTOCOL FOR THE 1997 DIAZINON AMBIENT MONITORING IN FRESNO COUNTY

Enclosed is the final protocol, "Protocol for the Ambient Air Monitoring of Diazinon in Fresno County During Winter, 1997."

If you or your staff have questions or need further information, please contact me at (916) 263-1630 or Mr. Kevin Mongar at (916) 263-2063.

Enclosure

cc: Genevieve Shiroma, Chief (w/Enclosure)
Air Quality Measures Branch
Stationary Source Division

State of California
California Environmental Protection Agency
AIR RESOURCES BOARD

Protocol for the Ambient Air Monitoring of Diazinon In Fresno County During Winter, 1997

Engineering and Laboratory Branch

Monitoring and Laboratory Division

Project No. C96-036

Date: January 9, 1997

APPROVED:

Kevin Mongar, Project Engineer

Cynthia L. Castronovo, Manager
Testing Section

George Lew, Chief
Engineering and Laboratory Branch

Protocol for the Ambient Air Monitoring
of Diazinon
In Fresno County During Winter, 1997

I. Introduction

At the request of the California Department of Pesticide Regulation (DPR), (October 31, 1995 Memorandum from John Sanders to George Lew) the Air Resources Board (ARB) staff will determine airborne concentrations of the pesticide diazinon [O,O-Diethyl O-(6-methyl-2-(1-methylethyl)-4-pyrimidinyl) phosphorothioate] over a six week ambient monitoring program in areas frequented by people. This monitoring will be done to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5) which requires the ARB "to document the level of airborne emissions .... of pesticides which may be determined to pose a present or potential hazard..." when requested by the DPR. The monitoring program will be conducted in Fresno County.

The draft method development results and "Standard Operating Procedures for the Analysis of diazinon in Ambient Air" are not included in this protocol but will be included in the draft report.

II. Chemical Properties of Diazinon

Pure diazinon (CAS:333-41-5) is a clear colorless liquid with a faint ester-like odor. Technical grades are yellow. Diazinon has a molecular formula of $C_{12}H_{21}N_2O_3PS$, a formula weight of 304.35 g/mole, and a specific density of 1.116-1.118 at 20 °C. It has a water solubility of 71.1, 53.5, and 43.7 mg/L at 10, 20, and 30 °C respectively, a Henry's Constant of 1.13×10^{-7} atm·m^3/mol at 20 °C, and a vapor pressure of 8.47×10^{-5} mmHG at 20 °C. Diazinon is miscible with a variety of organic solvents.

The hydrolysis half-life ($t_{1/2}$) of diazinon in water (20°C) is 11.8 hours (pH 3.1); 185 days (pH 7.4); 136 days (pH 9.0) and 6 days (ph 10.4). Reported soil half-lives following incubation of 10 ppm diazinon are 12.5 weeks (sterile sand loam); 6.5 weeks (sterile organic soil); <1 week (non-sterile sand loam); and 2 weeks (non-sterile organic soil). Exposure of diazinon to UV light produces hydroxydiazinon. The photolytic $t_{1/2}$ for this reaction, in aqueous buffer solution (25 °C and pH 7.0), has been calculated to be 15 days. The $t_{1/2}$ of diazinon is approximately 3.2 weeks in a neutral solution at room temperature. Diazinon and its oxidative product diazoxon, have been found in fogwater. The distribution of diazinon (1.6 ng/m^3) was 76.1% (vapor phase); 19.8% (dissolved phase); 3.7% (air particles); and 0.4% (water particles). The distribution of diazoxon was 13.4%, 81.7%, 4.9%, and 0.02% respectively.

The acute oral LD_{50} of diazinon for rats ranges from 240 to 480 mg/kg. The LC_{50} (96 hour) for rainbow trout is 16 mg/L, and 2.6 to 3.2 mg/L for bluegill sunfish. The OSHA 8-hour time weighted average for a personal exposure limit is 0.1 mg/m^3. Diazinon has entered the risk assessment process at DPR under the SB 950 (Birth Defect Prevention Act of 1984) based on its potential for reproductive and mutagenic adverse health effects.

III. Sampling

Samples will be collected by passing a measured volume of ambient air through XAD-2 resin. The exposed XAD-2 resin tubes (SKC #226-30-06) are stored in an ice chest (dry ice) or freezer until desorbed with 3 ml of ethyl acetate. The flow rate will be accurately measured and the sampling system operated continuously with the exact operating interval noted. The resin tubes will be protected from direct sunlight and supported about 1.5 meters above the ground during the sampling period. At the end of each sampling period, the tubes will be capped and placed in culture tubes with an identification label affixed. Any diazinon present in the sampled ambient air will be captured by the XAD-2 adsorbent. Subsequent to sampling, the sample tubes will be transported on dry ice, as soon as reasonably possible, to the ARB Monitoring and Laboratory Division, Testing Section laboratory for analysis. The samples will be stored in the freezer or analyzed immediately.

A sketch of the sampling apparatus is shown in Attachment A. Calibrated rotameters will be used to set and measure sample flow rates. Samplers will be leak checked prior to and after each sampling period with the sampling cartridges installed. Any change in the flow rates will be recorded in the field log book. The field log book will also be used to record start and stop times, sample identifications and any other significant data.

Ambient Monitoring

The use patterns for diazinon suggest that monitoring should occur over a 30- to 45-day sampling period in Fresno County during the months of January or February. Three to five sampling sites should be selected in relatively high-population areas or in areas frequented by people. Sampling sites should be in almond and/or stone fruit growing areas but should not be immediately adjacent to fields where diazinon is being applied. At each site, twenty to thirty discrete 24-hour samples should be taken during the sampling period. Background samples should be collected in an area distant to diazinon applications.

Replicate (collocated) samples are needed for five dates at each sampling location. The date chosen for replicate samples should be distributed over the entire sampling period. They may, but need not be, the same dates at every site.

Four sampling sites plus an urban background site were selected by ARB personnel from the areas of Fresno County where stone fruit farming is predominant. Sites were selected for their proximity to the orchards with considerations for both accessibility and security of the sampling equipment. The five sites were at the following locations: Addresses for the sites are listed in Table 1.

TABLE 1. Ambient Sampling Sites		
REE	Kings Canyon Unified District Office 675 W. Manning Reedley, CA 63654	(209) 637-1200 Carl Campbell
ARB	Air Resources Board, Ambient Air Monitoring Station 3425 N First, Suite 205B Fresno, CA 228-1825	(209) 228-1825
CEN	Centerville 48 S. Smith Centerville, CA 93657	(209) 787-2511 Rosemary Debillar, Principal
SAN	Fairmont Elementary School 3095 N. Greenwood Sanger, CA 93657	Kathy Hushek, Principal
PAR	Parlier High School 601 3rd Street Parlier, CA 93648	(209) 646-3573 Juan Sandoval, Principal

The samples will be collected by ARB personnel over a seven week period from January 13 - February 28, 1997. 24-hour samples will be taken Monday through Friday (4 samples/week) at a flow rate of approximately 2 L/minute.

IV. Analysis

The method development results and "Standard Operating Procedures for the Analysis of Diazinon in Ambient Air" are not included in this draft protocol but will be included in the draft report.

V. Quality Assurance

Field Quality Control for the ambient monitoring will include:

1) Five field spikes (same environmental and experimental conditions as those occurring at the time of ambient sampling), spiked at five different levels. The field spikes will be obtained by sampling ambient air at the background monitoring site for 24 hour periods at 2 L/minute.

2) Five trip spikes will be prepared and spiked at five different levels.

3) Replicate samples will be taken for seven dates at each sampling location.

4) Trip blanks will be obtained at each of the five sampling locations. Procedures will follow ARB's "Quality Assurance Plan for Pesticide Monitoring" (Attachment B).

The instrument dependent parameters (reproducibility, linearity and minimum detection limit) will be checked prior to analysis. A chain of custody sheet will accompany all samples. Rotameters will be calibrated prior to and after sampling in the field.

VI. Personnel

ARB personnel will consist of Kevin Mongar (Project Engineer) and an Instrument Technician.

APPENDIX II

DPR's MONITORING RECOMMENDATIONS FOR DIAZINON

State of California

Memorandum

To : George Lew, Chief
Engineering and Laboratory Branch
Monitoring and Laboratory Division
Air Resources Board
600 North Market Boulevard
Sacramento, California 95812

Date : October 31, 1995

Place :

From : Department of Pesticide Regulation — 1020 N Street, Room 161
Sacramento, California 95814-5624

Subject : MONITORING RECOMMENDATION FOR DIAZINON

Attached is the Department of Pesticide Regulation's recommendation for monitoring the insecticide diazinon. This recommendation is made pursuant to the requirements of Assembly Bills 1807 and 3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5). As you know, monitoring recommendations are made from historical use information for the pesticide in question. For this reason, it is essential that the agricultural commissioner, of the county where monitoring will be performed, be consulted prior to the onset of air monitoring.

We anticipate the submission of air monitoring data by March, 1997.

If you have any questions please contact Kevin Kelley, of my staff, at (916) 324-4187.

John L. Sanders

John S. Sanders, Chief
Environmental Monitoring and
Pest Management Branch
(916) 324-4100

attachment

cc: Paul H. Gosselin, DPR
Ronald J. Oshima, DPR
Gary T. Patterson, DPR
Charles M. Andrews, DPR
John Donahue, DPR
Barry Cortez, DPR
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Cara Roderick, ARB

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SURNAME			

State of California
Department of Pesticide Regulation
1020 N Street
Sacramento, California 95814-5624

Staff Report

USE INFORMATION AND AIR MONITORING RECOMMENDATION FOR THE PESTICIDAL ACTIVE INGREDIENT DIAZINON

October 1995

Principal Author

Kevin C. Kelley
Associate Environmental Research Scientist

MONITORING RECOMMENDATION FOR DIAZINON

BACKGROUND

In order to fulfill the requirements of AB 1807/3219 (Food and Agricultural Code, Division 7, Chapter 3, Article 1.5), the Department of Pesticide Regulation (DPR) has previously requested that the Air Resources Board (ARB) document the airborne concentrations of the pesticide diazinon [O,O-Diethyl O-(6-methyl-2-(1-methylethyl)-4-pyrimidinyl) phosphorothioate]. This recommendation provides background and recent use information on diazinon containing products, and identifies how they are used.

Pure diazinon (CAS: 333-41-5) is a clear colorless liquid with a faint ester-like odor. Technical grades are yellow. Diazinon has a molecular formula of $C_{12}H_{21}N_2O_3PS$, a formula weight of 304.35 g/mole, and a specific density of 1.116-1.118 at 20 °C. It has a water solubility of 71.1, 53.5, and 43.7 mg/L at 10, 20, and 30 °C respectively, a Henry's Constant of 1.13×10^{-7} atm·m³/mol at 20 °C, and a vapor pressure of 8.47×10^{-5} mmHg at 20 °C. Diazinon is miscible with a variety of organic solvents.

The hydrolysis half-life ($t_{1/2}$) of diazinon in water (20 °C) is 11.8 hours (pH 3.1); 185 days (pH 7.4); 136 days (pH 9.0) and 6 days (pH 10.4). Reported soil half-lives following incubation of 10 ppm diazinon are 12.5 weeks (sterile sand loam); 6.5 weeks (sterile organic soil); <1 week (non-sterile sand loam); and 2 weeks (non-sterile organic soil). Exposure of diazinon to UV light produces hydroxydiazinon. The photolytic $t_{1/2}$ for this reaction, in aqueous buffer solution (25 °C and pH 7.0), has been calculated to be 15 days. The $t_{1/2}$ of diazinon is approximately 3.2 weeks in a neutral solution at room temperature. Diazinon and its oxidative product diazoxon, have been found in fogwater. The distribution of diazinon (1.6 ng/m³) was 76.1% (vapor phase); 19.8% (dissolved phase); 3.7% (air particles); and 0.4% (water particles). The distribution of diazoxon was 13.4%, 81.7 %, 4.9%, and 0.02% respectively.

The acute oral LD_{50} of diazinon for rats ranges from 240 to 480 mg/kg. The LC_{50} (96 hour) for rainbow trout is 16 mg/L, and 2.6 to 3.2 mg/L for bluegill sunfish. The OSHA 8-hour time weighted average for a personal exposure limit is 0.1 mg/m³. Diazinon has entered the risk assessment process at DPR under the SB 950 (Birth Defect Prevention Act of 1984) based on its potential for reproductive and mutagenic adverse health effects.

USE OF DIAZINON

As of September 1, 1995, there were 254 active registrations for products containing diazinon. These products consist of ear tags for cattle, flea and tick collars for pets, home use products for the control of lawn insects, and agricultural products. Formulations of diazinon include impregnated plastics (flea collars, ear tags), granular, aqueous and emulsifiable concentrates, wettable powders, dusts, flowables, microcapsules, and fumigants. The Signal Words on products

which contain 25% or more diazinon are "Warning"; products containing 15% or less (most home use products) are labeled "Caution".

Diazinon use for 1993, 1992, and 1991 is summarized in the following tables: Table 1, Diazinon Use by Year; and Table 2, Diazinon Applications in Fresno County. Agricultural use of diazinon for the eleven counties listed in Table 1 accounts for 50 to 65% of total diazinon use. The remaining 50 to 35% of use is split fairly evenly between agricultural use (in counties not listed in table 1), and non-agricultural use (structural pest control, landscape maintenance, pet, and livestock products).

Table 1. Diazinon Use by Year (Pounds of Active Ingredient)

County	1993	1992	1991
Butte	73,471.1	48,689.7	31,531.7
Fresno	219,186.5	168,455.9	135,231.5
Imperial	56,015.7	58,023.1	76,784.5
Kern	99,782.7	93,528.8	46,674.7
Madera	57,010.6	70,221.9	52,411.4
Merced	43,664.6	60,883.9	31,968.0
Monterey	65,637.4	44,090.8	36,527.8
San Joaquin	46,929.3	54,465.8	26,974.9
Stanislaus	85,818.4	54,444.7	25,682.1
Sutter	49,905.9	50,008.9	19,518.6
Tulare	120,432.2	50,145.6	31,544.5
County Totals	917,854.4	752,959.1	514,849.7
CALIFORNIA TOTAL	1,491,709.59	1,347,155.52	1,007,021.85

The data summarized in table 1 show that the largest applications of diazinon routinely occur in Fresno County. Additionally, these data show that the greatest applications generally occur during January and February of each year (Table 2).

Diazinon is used as a dormant spray for the control of Lepidopterous insects, scale, mites, and aphid pests of fruit and nut trees. Diazinon is also applied during the spring for the control of different Lepidopterous insects, but at rates much lower than dormant spray rates. Dormant rates range from 3.9 lbs AI/acre (plums,) to 2.1 lbs AI/acre (apricots). Diazinon is also applied during this period (January–February) to cherries, apples, lettuce, walnuts, and greenhouse or nursery plants. Diazinon use on these commodities is minimal compared with applications to plums, almonds, peaches, and nectarines.

Table 2. Diazinon applications in Fresno County (Pounds of Active Ingredient)

Fresno County	1993	1992	1991
January (lbs AI)	73,717.7	38,375.2	17,731.7
(Rate)	2.7	1.94	2.22
February (lbs AI)	40,617.2	17,199.2	28,214.9
(Rate)	2.12	1.66	4.02

RECOMMENDATIONS

Ambient Air Monitoring

The use patterns for diazinon suggest that monitoring should take place in Fresno County during a 30– to 45–day sampling period in the months of January or February. Three to five sampling sites should be selected in relatively high-population areas or in areas frequented by people. Sampling sites should be in almond and/or stone fruit growing areas but not immediately adjacent to fields to which diazinon is being applied. At each site, twenty to thirty discrete 24-hour samples should be taken during the sampling period. Background samples should be collected in an area distant to diazinon applications.

Replicate (co-located) samples are needed for five dates at each sampling location. Two co-located samplers (in addition to the primary sampler) should be run on those days. The date chosen for replicate samples should be distributed over the entire sampling period. They may, but need not be, the same dates at every site. Field blank and spike samples should be collected at the same environmental (temperature, humidity, exposure to sunlight) and experimental (air flow rates) conditions as those occurring at the time of ambient sampling. Since diazinon is known to partition into fogwater, samples collected during fog episodes should be designated as such.

Monitoring of an Application Site

The use pattern for diazinon suggests that application-site monitoring should be conducted during the months of January or February in Fresno County, and that the monitoring be associated with applications of diazinon to almonds or stone fruits. Due to the extensive use of diazinon on these crops during this period, care should be taken so that other applications to nearby groves during the sampling period do not affect sample collection. A three day monitoring period should be established with sampling times as follows: Application + 1 hour,

followed by one 2-hour sample, one 4-hour sample, two 8-hour samples, and two 24-hour samples. A minimum of four samplers should be positioned, one on each side of the field. A fifth sampler should be co-located at one position. Since diazinon is extensively used in the area, background samples should collect enough volume (either 12 hours at 15 liters/min., or a shorter period with a higher volume pump) to permit a reasonable minimum detection level. Ideally, samplers should be placed a minimum of 20 meters from the field. Field blank and field spike samples should be collected at the same environmental (temperature humidity, exposure to sunlight) and experimental (similar air flow rates) conditions as those occurring at the time of sampling.

We also request that you provide in the monitoring report: 1) An accurate record of the positions of the monitoring equipment with respect to the field, 2) an accurate drawing of the monitoring site showing the precise location of the meteorological equipment, trees, buildings, etc., 3) meteorological data collected at a minimum of 15 minute intervals including wind speed and direction, humidity, and comments regarding degree of cloud cover, and 4) the elevation of each sampling station with respect to the field, and the orientation of the field with respect to North (identified as either true or magnetic north). Samples collected during fog episodes should be designated as such.

APPENDIX III

STANDARD OPERATING PROCEDURES FOR THE SAMPLING AND ANALYSIS OF DIAZINON IN AMBIENT AIR

State of California
Air Resources Board
Monitoring and Laboratory Division/ELB

Standard Operating Procedure for the Sampling and Analysis of Diazinon in Ambient Air

January 1997

1. SCOPE

This is a sorbent tube, solvent extraction, gas chromatography/mass selective detector method for the determination of diazinon from ambient air samples.

2. SUMMARY OF METHOD

The exposed XAD-2 resin tubes (SKC #226-30-06) are stored in an ice chest on dry ice or freezer until desorbed with 2.5 ml of ethyl acetate. The splitless injection volume is 1 ul. A gas chromatograph with a DB-35 capillary column and a mass selective detector is used for analysis.

3. INTERFERENCES/LIMITATIONS

Method interferences may be caused by contaminants in solvents, reagents, glassware and other processing apparatus that can lead to discrete artifacts or elevated baselines. A method blank must be done with each batch of samples to detect any possible method interferences.

4. EQUIPMENT AND CONDITIONS

A. INSTRUMENTATION:

Hewlett Packard 5890 chromatograph
Hewlett Packard 5971A mass selective detector
Hewlett Packard Varian 8200 Autosampler

Detector: 280°C
Injector: 285°C
Column: J&W Scientific DB-35, 30 meter, 0.25 mm i.d., 0.25 um film thickness.

GC Temp. Program: Initial 77°C, hold 2 min, to 170°C @ 55°C/min., to 205 C @ 3 C/min., to 250 C @ 70 C/min., hold 5 min.

Gas Flows:
column: He, 1.0 mL/min (9.2 psi @ 77 C), electronic pressure control

B. AUXILIARY APPARATUS:

1. Glass amber vials, 8 mL capacity.
2. Vial Shaker, SKC, or equiv.
3. Autosampler vials with septum caps.

C. REAGENTS

1. Ethyl Acetate, Pesticide Grade, or better
2. Diazinon, 98% pure or better (e.g., from Chem Service).
3. Diazinon D-10, 98% pure or better (e.g., from Cambridge Isotope Laboratories)

5. ANALYSIS OF SAMPLES

1. It is necessary to analyze a solvent blank with each batch of samples. The blank must be free of interferences. A solvent blank must be analyzed after any sample which results in possible carry-over contamination.

2. If a standard curve is not generated each day of analysis, at least one calibration sample must be analyzed for each batch of ten samples. The response of the standard must be within 10% of previous calibration analyses.

3. Carefully score the primary section end of the sampled XAD-2 tube above the retainer spring and break at the score. Remove the glass wool plug from the primary end of the XAD-2 tube with forceps and place it into an 8 mL amber colored sample vial. Pour the XAD-2 into the vial and add 2.5 mL ethyl acetate. Retain the secondary section of the XAD-2 tube for later analysis if needed to check the possibility of breakthrough.

4. Place the sample vial on a desorption shaker (or ultra sonic water-bath) for 30 minutes. Remove the diazinon extract and store in a second vial at -20°C until analysis.

5. After calibration of the GC system, inject 1.0 ul of the extract. If the resultant peaks for diazinon have a measured area greater than that of the highest standard injected, dilute the sample and re-inject.

6. Calculate the concentration in ng/mL based on the data system calibration response factors. If the sample has been diluted, multiply the calculated concentration by the dilution factor.

7. The atmospheric concentration is calculated according to:

Conc., ng/m^3 = (Extract Conc., ng/mL X 2.5 mL) / Air Volume Sampled, m^3

6. QUALITY ASSURANCE

A. INSTRUMENT REPRODUCIBILITY

Triplicate injections of 1 uL each were made of diazinon standards at four concentrations in order to establish the reproducibility of this instrument. This data (Testing Section lab, 2/11/97) is shown in Table 1.

TABLE 1
Instrument Reproducibility

D-10 Amt. (ng/ml)	D-16 Response	Diazinon Amt. (ng/ml)	Diazinon Response	Amt. Ratio	Resp Ratio	Response Ratio RSD
960	6902	129	724	0.134	0.105	
960	5750	129	621	0.134	0.108	
960	5150	129	589	0.134	0.114	4.2%
960	6450	258	1497	0.269	0.232	
960	6451	258	1448	0.269	0.225	
960	6610	258	1543	0.269	0.233	1.9%
960	4838	516	2530	0.538	0.523	
960	5048	516	2596	0.538	0.514	
960	5406	516	2739	0.538	0.507	1.6%
960	5495	1033	5976	1.08	1.09	
960	5768	1033	6138	1.08	1.06	
960	5928	1033	6316	1.08	1.07	1.4%

B. LINEARITY

A four point calibration curve was made (Testing Section lab, 2/11/97) ranging from 129 ng/mL to 1033 ng/mL diazinon (from TABLE 1). The corresponding linear regression equation and correlation coefficient are:

Response Ratio = (1.03)(Amount Ratio) - 0.038 Corr. Coef. = .9995

where:

Response Ratio = (diazinon response)/(diazinon D-10 response)

Amount Ratio = (diazinon concentration)/(diazinon D-10 concentration)

C. MINIMUM DETECTION LIMIT

Using the equations and data above, the limit of detection (LOD) and the limit of quantitation (LOQ) for diazinon were calculated by:

$$LOD = |A| + 3(S)$$

where:

|A| = the absolute value of the x-intercept of the standard curve (from above).

(S) = the relative standard deviation of the responses of the lowest concentration used for the standard curve times A (RSD x A).

LOD = |0.0369| + 3(0.0369)(.0443) = 0.0418 (response ratio)
= 40.1 ng/ml diazinon

LOQ = 3.3(LOD) = 132 ng/ml

Based on the 2.5 mL extraction volume and assuming a sample volume of 2.88 m^3 (2 lpm for 24 hours):

(132 ng/mL)(2.5 mL) / (2.88 m^3) = 115 ng/m^3 per 24-hour sample

D. COLLECTION AND EXTRACTION EFFICIENCY (RECOVERY)

Five microliters of a 106.9 ng/ml diazinon standard were spiked on the primary section of each of four XAD-2 sampling tubes. The spiked tubes were then subjected to an air flow of 2 lpm for 24 hours. The samplers were set-up in a garage/shop at an ambient temperature of approximately 85°F (maximum). The primary and back-up sections were then separately desorbed with ethyl acetate and analyzed. Percent recoveries of diazinon from primary sections of the four tubes were 98.8%, 101%, 96.5% and 98.9% with an average of 98.8%.

E. STORAGE STABILITY

Storage stability studies were conducted over a 125 day period. The primary sections of eight tubes were spiked with 4500 ng of diazinon. The spiked tubes were stored in the freezer at -20 C and extracted/analyzed on storage days 19, 49 and 125. Two tubes each were analyzed on days 19 and 49 while 3 tubes were analyzed on day 125 (1 tube was lost during extraction). The storage recoveries (average results) were 97.5%, 81.9% and 90.7% for days 19, 49 and 125 respectively.

F. BREAKTHROUGH

The primary sections of four tubes were spiked with 534.5 ng diazinon/tube then run for 24 hours at 2 lpm (see Section D above). No diazinon was detected in the back-up resin bed of any of the tubes.